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ONTARIO HIGH SCHOOL

LABORATORY MANUAL IN CHEMISTRY

Authorized by

The Minister of Education for Ontario

Price 20 cents

TORONTO

THE MACMILLAN COMPANY OF CANADA, LTD.

1915



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First edition, printed August, 1909

Reprinted February, 1910.

1913

CHEMISTRY MANUAL

INTRODUCTION

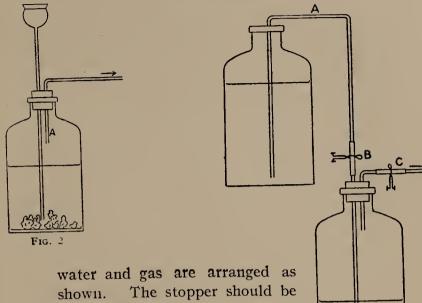
As this Manual is intended for use in secondary schools of all grades, it has been thought best to leave to individual teachers the selection of the experiments which the class shall be required to do and those which the teacher himself will use for demonstration purposes. The equipment of the schools, the experience of the teachers, and the exigencies of the time-table, are so variable that it seems unwise to prescribe a division which, suitable in some cases, would be quite unsatisfactory in others. Most of the experiments should always be done by the pupils, some may be done by either pupils or teacher, and a few probably would, for various reasons, be better performed by the teacher. These last have been retained in the Text-book, the others are in the Manual.

According to the general practice of the best authors and teachers, questions have been appended to the experiments to fix the learner's attention on the problem to be determined, otherwise confusion of mind is certain to arise through the heed of non-essentials.

As this book is likely to be used by many teachers who have not had experience in working through a systematic course of experiments, the following suggestions are offered in the hope that they make the work easier for such teachers and more profitable for the learners.

(1) Use *small* quantities of materials, unless when otherwise stated.

- (2) Add reagents gradually, a direction to pour a liquid into a vessel does not mean to dash a lot of 't in.
- (3) Never bring a flame near a hydrogen reservoir or a delivery tube carrying hydrogen until the gas has been tested. This applies equally to acetylene.
- (4) Always cut phosphorus under water and handle it with forceps. Burn any scraps or residue that may remain from cutting the substance or from an experiment. Keep it under water in a glass-stoppered bottle or jar.
- (5) Never smell escaping gas rashly. A safe plan is to hold the hand half-closed over the mouth of the vessel or of the delivery tube, then smell the gas in the hand.
- (6) Metal deflagrating spoons are generally unsatisfactory; cups made of chalk or of plaster of paris are more serviceable.
- (7) Floats to carry such cups or other material may be made of a flat cork, loaded with lead on the under side.
- (8) Corks should be filled with vaseline, and rubber stoppers that have been in use for some time will generally permit gas to leak unless coated with vaseline. Ground stoppers are also made more secure by this treatment.
- (9) Figure 1 shows a convenient arrangement for the storage of gas, such as oxygen, if regular gas holders are not available. A half-gallon bottle (a Winchester, which may be had for a few cents at a drug store) is set on a shelf or stand and from it water may be siphoned into another bottle, B, filled with the gas; clips to control the flow of



vaselined both on the outside and at the perforations.

(10) For the preparation of Fig. 1 hydrogen and other gases that do not require to be heated, a bottle is a serviceable generator (Fig. 2). In the preparation of hyd-

rogen, a safety tube should be used. In the figure the funnel tube serves this purpose and permits new acid to be introduced.

(11) Bottles of about 4 oz. capacity serve very well as test tubes and gas holders, when heat has not to be applied to them. Test tubes, beakers and flasks are frail and are liable to become expensive through breakage, hence the usefulness of bottles.

(12) Soup plates are very convenient pieces

of apparatus, especially when collecting gases.

(13) Only few schools have proper fume chamber accommodation for class work. Windows may be utilised for this purpose by having boxes constructed of dimensions a foot wide, fifteen inches high and length equal to the breadth of the window; front and back should be of glass, the front hinged at the top side. This box, when in use, may be set on the window sill, the sash raised until it rests on top of the box, and the latter shoved outward until a slit left in the cover is outside of the sash.

(14) Pupils and teacher should be provided with protective aprons, preferably of rubber, because accidents will happen where glassware is being heated and acids used. These aprons should extend from the neck to the feet so as to save the clothing from injury in case fluids spatter.

With regard to the teacher's work the following suggestions are offered, but only as suggestions, because every teacher, whether experienced or not, must do his best work in his own way:

- (1) Keep a note-book, properly indexed, in which will be entered references, information and points of difficulty, either of matter or treatment.
- (2) Read standard text-books on Chemistry, and on methods and aims of science teaching.
 - (3) Plan work ahead to save time.
- (4) At times it will not only be practicable, but desirable, to have different pupils, or different groups, work at various parts of a problem at the same time, and report results. As an example, there is very little profit to the student from the repetition, but much loss of good time, if every member of a class determines whether or not hydrogen is set free by the action between zinc, magnesium, iron and copper separately and each of the three common acids. Two cases are educationally as good as twelve, and five-sixths of the time is saved.

- (5) Concentrate the pupils' attention on the problem in hand until it is solved; then any side issues that are worth while may be taken up.
- (6) A fact is valuable only in its relationships, so an experiment should be done only because it leads to something of importance in the scientific system, not because it is interesting in itself.
- (7) Have notes written and results entered in the class at the time the experiment is made. Sketches of necessary apparatus are an essential part of such notes, verbal descriptions are not sufficient. Notes should be written in ink, and corrections made without erasure. This will lead at first to some untidiness, but the aim of the study is educational progress, not the production of pretty and artistic exercise books.

CHAPTERS I and II

MATTER AND ENERGY

Physical and Chemical Changes

1. Make a slit in the end of a match and in it place a piece of platinum wire two or three inches long; then, using the match as a handle, hold the wire in the flame of a lamp or gasburner.

Note any changes that occur. After heating the wire for a couple of minutes, remove it from the flame and let it cool. Has it changed? Was there any alteration during the heating? Is it still platinum?

Repeat the experiment but use magnesium wire.

Compare the changes with those that occurred in the case of platinum. Is the material still magnesium?

2. Boil some water in a tt. ntted with a delivery tube that leads into a cold, dry flask.

When the water boils, what passes out of the delivery tube? Where does it come from? What becomes of it? Is it the same material at the end of the operation that it was at the beginning?

Put some water, to which a few drops of sulphuric acid have been added, into an electrolytic apparatus until the tubes are full; then close the tubes and turn on the current.

Is it steam that gathers in the tubes?

After a time, open the one that has most gas in it, while holding a lighted match at its mouth. Do the same thing with the other, but let the match be only glowing. Compare the results with those noted when water was

boiled. Observe particularly the characteristics and sources of the different kinds of matter obtained.

3. Dissolve some sugar in water, taste the water. Evaporate a little of it to dryness.

What is left? In what condition was the sugar when dissolved? Did it change to anything else than sugar?

Carefully weigh out about a gram of sugar, and heat it on a piece of tin or mica until it ceases to burn.

What is the appearance of the substance that is left? Has it changed in weight; if so, to what extent? What taste has the residue? Is it sugar? Given that weight is the measure of the quantity of matter in a body, is there as much matter in the black residue as there was in the sugar?

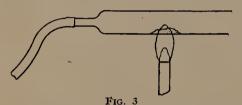
4. Select a hard glass test tube about a quarter of an inch in diameter and put a little red oxide of mercury into it; this may be done without smearing the glass by rolling a strip of paper into a tube, thrusting it down inside the glass one, and pouring the powder through it. Withdraw the paper, then heat the oxide to redness in the glass tube. When a mistiness begins to show on the glass, hold a glowing splinter in the tube. Continue the heating until distinct globules are formed on the walls of the tube, then turn the latter mouth downward and strike it sharply on a piece of clean paper on the table to jar out any loose matter in it.

This experiment is intended to determine the following points: (1) Does oxide of mercury undergo any permanent change when heated? (2) Does it yield a kind, or kinds, of matter different from itself? What are your answers to these questions? Upon what observations are your answers based?

5. Put flowers of sulphur in a tt. to a depth of about

an inch; heat the tube gradually, turning it about and moving it in and out of the flame. When vapour begins to come off, hold a cold bottle inverted over the mouth of the tube. Note the changes that take place in the appearance and condition of the sulphur, and make determinations regarding sulphur similar to those previously made about oxide of mercury.

6. Put two grams of fine copper filings, or the copper dust obtained by reducing the oxide, into a piece of hard glass tubing about four inches long and a quarter of an



inch inside diameter, balance the tube and contents on a scale; and, without removing the weights, lift off the tube and attach it to an oxygen tank.

Heat the tube to redness, letting a slow stream of oxygen pass through it. When the copper has changed to a black mass, disconnect the tube, let it cool and put it back on the scale-pan from which it was taken.

Has the weight changed? Turn the substance in the tube out on a piece of paper and examine it carefully. What two kinds of matter were in the tube? Does the black substance contain other matter than copper?

Put the black powder back into the tube, and, in case any of it has been lost, balance it again on the scale; then, by means of a rubber connection, attach the tube to a gas tap; heat it as before but allow coal gas to pass slowly over it. Keep the gas burning at the mouth of the tube while the powder is being heated, and until the tube cools. Continue the heating for four or five minutes, then let the tube cool and replace it on the scale.

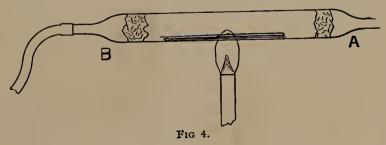
How has the weight been affected? What is now in the tube?

For those schools in which coal gas is not available the experiment may be varied by heating the black powder with twice its bulk of powdered charcoal in a glass tube, closed at one end. The tube should be kept red-hot for five minutes, then allowed to cool. Afterwards the contents may be scraped out into an evaporating dish and the charcoal washed away by filling the dish several times with water and allowing the latter to flow out over the lip. The metal will now be left at the bottom of the dish. The final weighing should be omitted, for loss of material is likely to occur in the operation of washing.

7. Powder some lead nitrate and put about a gram of it in a glass tube about four inches long, closed at one end; balance it on a scale and leave the balancing weights on the pan. Heat the tube to redness for about five minutes, and, when it is cold, put it on the same scale-pan as before. Scrape the residue out of the tube on paper.

Compare what is left with the substance originally put in the tube. What does the change of weight indicate about the quantity of matter in the tube at the end of the operation as compared with that at the beginning?

8. Select a piece of glass tubing about half an inch in diameter, draw the end out as at A, and put a loose plug of asbestos in it. Fold up about a foot of magnesium rib-



bon and put it in the tube with another asbestos plug behind it at B. If necessary, draw out the end of B so that a rubber connection will fit over it. Balance the whole upon a scale, leave the balancing weights on the pan, and attach the tube to the oxygen tank. Heat the tube to redness while oxygen is passing slowly through it. While the magnesium is burning no white smoke should escape through the asbestos. When the metal has burned, detach the tube, let it cool and weigh it again.

Cut the tube in two and examine the white powder. Compare it with the substance obtained in Experiment 1. Is there any change in the quantity of matter in the white powder as compared with the quantity of matter in the magnesium alone? What matter made up this increase of weight? Compare this result with that of Experiment 4.

CHAPTER III

ELEMENTS AND COMPOUNDS

9. Weigh a piece of zinc of about 4 grams in weight, put it in a beaker and add 1 c.c. of sulphuric acid for every two grams of metal; then pour in slowly, with constant stirring about, twice the volume of water that there is of acid. When gas ceases to rise, take out the remaining zinc, wash it, dry it and weigh it; evaporate the liquid left in the beaker to dryness. The white solid is a compound of zinc and sulphuric acid.

The points to be noted are the loss of weight of the zinc due to the disappearance of some of the metal, and the formation of a new kind of matter.

CHAPTER IV

MIXTURE, SOLUTION AND COMPOUNDS

10. Weigh out 4 grams of powdered sulphur and 7 grams of clean iron filings, mix the two thoroughly, and notice if particles of iron and of sulphur are perceptible with a lens. Make a pile of part of the mixture on a sheet of paper and draw a magnet through it; tap the magnet over the pile to shake off any free sulphur, then pick off the filings and lay them on one side; repeat this as often as any appreciable quantity of iron sticks to the magnet.

Shake some of the original mixture with water in a tt. and allow the suspended matter to settle. Is the sediment uniform, as the mixture was? Shake up the sediment and pour off the water with its suspended matter; repeat this two or three times; what remains in the tt.?

Put a little of the original mixture in a tt. and pour carbon bisulphide over it until the powder is well covered; cork the tube and allow it to stand for ten minutes, then pour off the clear liquid into a dish and let it evaporate under the hood.

Treat a little of the mixture in a tt. with hydrochloric acid, smell the gas that comes off; when all action ceases, add a little more acid, and if no more gas is formed, notice if any sediment remains.

Place enough of the mixture of iron and sulphur in a hard glass test tube to fill it for an inch or more; hold the very end of the tube in the flame until the mixture begins to glow, then remove it from the flame; after all action has ceased and the mass has cooled, break the tube and examine the substance inside of it. This experiment may be varied by using fine copper filings instead of iron; the proportions should then be two of copper to one of sulphur.

Powder the material that came out of the tube, and repeat the experiments with the magnet, water, carbon bisulphide and hydrochloric acid.

The object of this experiment is to determine if a new kind of matter having properties different from either the iron or sulphur has been produced.

Tabulate the results of your observations and state the conclusion you draw from them. Is there any evidence that energy, as either heat or light, has been given off?

11. Drop a very small crystal of permanganate of potash into a litre of water and stir until the crystal disappears. Lift out a drop of the water on the end of a glass rod.

Is there any permanganate in it? What conclusion may be drawn as to the state of division of the crystal?

12. To find whether one liquid is soluble in another: Let a drop of sulphuric acid fall into a clear solution of barium chloride, or barium nitrate, in a tt. This is the test for sulphuric acid:

Put about 10 c.c. of water in a vessel, then add four or five drops of sulphuric acid.

Taste the liquid by placing a drop on the tongue. Let a drop of the liquid fall into barium chloride solution. What is the answer to the problem that was to be solved? What warrants that answer?

13. To determine whether gases will dissolve in water: Fill a large flask with fresh cold water and let it stand quietly in a warm room for a couple of hours, but observe it every half hour or so.

The bubbles are composed of air. Notice: (1) When the bubbles appear; (2) If they change in size; (3) If they are on all parts of the vessel.

Why do the bubbles not appear at once? (Refer to Solubility of Gases in a book on Physics.)

14. Heat a little ammonia solution (hartshorn) in a tt. that is fitted with a delivery tube. Smell a handful of the gas that comes off. Invert a large tt. over the delivery tube and, after a couple of minutes, remove the inverted tube and set it mouth downward in water.

The gas in the tube must either condense or be absorbed. Are there conditions that would cause condensation to the extent noticed?

Dissolve a little copper sulphate, and let a drop of the original hartshorn fall into it. The colour gives a test for ammonia.

Turn the tt. that had the gas in it mouth upwards, retaining the water that has risen in it, and smell it. Pour a few drops of this water into copper sulphate solution.

Is ammonia soluble in water?

15. Select two gas cylinders or large test tubes of equal size; fill each with water, then choose two lumps of copper sulphate of about the same size, each as large as a small marble; drop one of these lumps into one cylinder, and hang the other in a piece of muslin, just below the top of the water in the second cylinder. Let them stand quietly over night.

What difference is there in results, as judged by colour? How is the difference accounted for?

Bichromate of potash, permanganate of potash or ferricyanide of potash may be used as alternatives for the copper sulphate in this experiment.

16. Put about one gram of sugar in each of three test tubes, pour on the sugar in one tube some coal oil, in another tube some water, and in the third some sulphuric acid; let them stand until next lesson.

How is the sugar affected in each case?

17. Stir into boiling water in a beaker powdered chlorate of potash until the powder just ceases to dissolve. Set the beaker aside to cool.

What happens? Is there any chlorate still dissolved in the water? Test this by evaporating a drop of the liquid on mica or tin. Where did the solid come from that is at the bottom of the beaker? What is the relation between temperature and the quantity of the chlorate dissolved by water?

CHAPTER V

HYDROGEN

Note.—When hydrogen is mixed with air it is dangerously explosive if ignited, hence care must be taken that a flame is not brought near such a mixture. When it is necessary to light a jet of hydrogen the gas must be tested to see that it is pure. This is done by collecting a tt. full of the gas over water and holding it to a flame. If the gas burns slowly and quietly, the danger is past. The hydrogen at the jet may safely be ignited when the flame of that burning in the tt. can be used to light it.

18. Put into a hydrogen generator some pieces of zinc, cover them with dilute sulphuric acid. When all air has been expelled (see note above), lead the gas through a fine metal tube, such as the nozzle of a blowpipe and ignite it. Hold a cold, dry bottle mouth downward over the flame.

What is formed inside of the bottle?

Collect several large test tubes full of the gas over water. Turn one mouth upward and quickly ignite the gas; watch for the flame in the tube. Turn another mouth upward and let it stand for a couple of minutes, then try to ignite it. Repeat the last experiment, but hold the tube mouth downward.

What appearance has the flame of hydrogen when burning in the wide tube? What conclusion follows from the effort to light the gas in the tube that stood mouth upwards?

Try if hydrochloric acid diluted one-half with water may be substituted for sulphuric in the preparation of hydrogen by the use of zinc. Try if copper, iron, lead and magnesium may be used instead of zinc. Wellocklike

The following two experiments are intended to illustrate those properties of hydrogen that relate to combustion:

- 19. Collect a large tt., or better a gas cylinder, full of hydrogen over water, then thrust a lighted splinter four or five inches up into the gas while the cylinder is held mouth downwards.
- 20. Collect a soda water bottle full of hydrogen over water and ignite the gas. Collect the same bottle one-third full, lift it out of water and allow air to enter, then hold the mouth of the bottle to a flame, but directed away from any person.

Repeat the last experiment with the following changes: Select a cork that will fit the bottle, bore a hole in it and put a piece of glass tubing through it; cut off the projecting ends of the tube. Fill the bottle with a mixture, one-third hydrogen and two-thirds air, and put the prepared cork in the mouth of it. Wrap the bottle in a towel and lay it so that if the cork blows out no one will be injured, then with a long, blazing splinter ignite the gas at the vent in the cork.

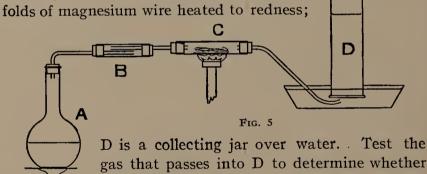
How is the pressure on the inside of the bottle affected by the burning of the gases?

Other methods of preparing hydrogen:

21. Fill an electrolytic apparatus with water, to which a few drops of sulphuric acid have been added. Connect

the battery and note the unequal volumes of the gases set free. Make a very fine nozzle by drawing out a piece of glass tubing and attach it to that branch of the apparatus in which most gas is collecting. When four or five inches of gas have accumulated in this tube allow it to escape through the nozzle, but at once ignite it and invert a cold, dry test tube over the flame. Examine the tube for condensed vapour. Allow more gas to form in the apparatus, collect a dry tube full of it and quickly hold the mouth of the tube to a flame. Examine the test tube for moisture, as before.

22. Fit up apparatus as figured. A is a flask in which water is boiling; B is a water trap, so that only steam may enter C; C is a hard glass tube containing



gas that passes into D to determine whether it is hydrogen. The collecting jar of Fig. 16 may be substituted for the one here shown. In that case, by depressing the jar, opening

the clip and igniting the gas, it may easily be shown if water is formed by inverting a cold, dry bottle over the flame. Using the same apparatus, substitute clean iron filings for magnesium and determine if hydrogen is given off.

23. In this experiment the water should be covered with a glass plate, or some of the molten material may be thrown to some distance,

Drop a small piece of clean sodium, not larger than a grain of wheat, on some water in a soup plate. Immediately cover the plate with a sheet of glass. Drop another similar piece on a scrap of filtering paper laid on the water. Boil a little water in an evaporating dish and put a third piece of sodium on it. Place a little piece of potassium on water.

Note the shape which the piece of metal takes, its state, movement and apparent ignition.

24. Prepare a glass tube two inches long and a quarter of an inch in diameter, closed at one end. Cut off a piece of sodium, rub it free from oil on filter paper and mould it into a pellet that will fit tightly into the open end of the tube; the metal should be flush with the mouth of the tube. Drop the whole into water on a plate and collect the escaping gas in a test tube over water.

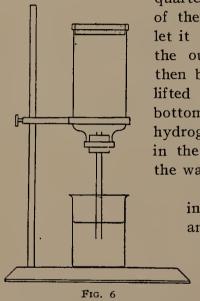
Test this gas to find if it is hydrogen.

25. Determine the solubility of hydrogen in the following way: Fill a eudiometer with cold water that has been boiled to expel all dissolved air, invert it, keeping its mouth under water, and pass in pure hydrogen to the depth of about two centimetres. Note accurately the volumes of gas and of water in the tube. Close the mouth of the tube carefully by placing the thumb over it and shake the water and gas together for a couple of minutes. Replace the tube with its mouth under water while it is still closed, but repeat the shaking four or five times in the course of an hour. Then read the volumes of gas and of water as before; but, if necessary, correct for change of temperature during the operation. (See H. S. Physics.)

What change has occurred in the volume of the gas? How many volumes of water were in the tube at first? How many volumes of gas were

dissolved? How many volumes of gas would 100 volumes of water dissolve?

26. Cast a plaster of paris bottom in a small tubulated bell jar. (An 8-oz. bottle with the bottom cut off evenly will answer very well). To do this proceed as follows: Rub some oil or vaseline on a piece of glass and flow a thin paste of plaster and water over this to a depth of a



quarter of an inch. Press the base of the jar down into the paste and let it stand until the plaster sets; the outside part of the sheet may then be trimmed away and the jar lifted off, and set aside until the bottom dries. Fill the jar with hydrogen, then mount it as shown in the diagram. Watch the level of the water in the tube.

Does the height of the water in the tube vary? Is this due to an alteration in the *volume*, or a change in the quantity of the gas in the jar?

Vary this experiment by making an explosive mixture

of hydrogen and air (about one-fourth hydrogen) in a bottle; then fill the jar with this mixture. Mount the jar as in the previous case, but let it stand for a quarter of an hour, then take out the cork and hold the mouth to a flame.

The property of gases, illustrated in the case of hydrogen by these experiments, is known as diffusibility (refer to the H. S. Physics under this head).

CHAPTER VI

OXYGEN

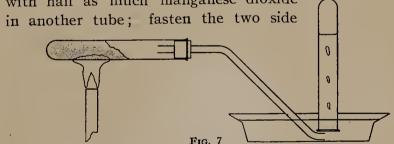
27. Repeat Experiment 4, but this time for the purpose of determining whether or not a gaseous substance passes out of the tube; and, if so, what effect it has on a burning splinter.

This gas, which causes a glowing splinter to glow more brightly, is Oxygen.

28. Heat a little powdered potassium chlorate in a hard glass tube closed at one end, and when the chlorate begins to boil hold a glowing splinter in the mouth of the tube. Drop a small bit of charcoal into the tube. Dissolve separately in distilled water a little unheated chlorate and some of the white solid left in the tube; let a drop of silver nitrate solution fall into each.

Has the substance undergone a chemical change when heated? Describe the changes that took place in the chlorate when heated?

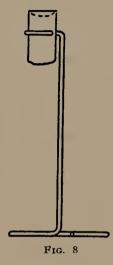
29. Put about an inch in depth of powdered potassium chlorate in one tt. Mix the same quantity of chlorate with half as much manganese dioxide



by side; heat both equally. Test from time to time with a glowing splinter.

From which one does oxygen pass off first? Which yields oxygen with the less heating?

30. Mix some powdered chlorate of potash with half its bulk of manganese dioxide. Heat some of this in a hard glass tt. that is fitted with a delivery tube. Arrange the apparatus, as in Fig. 7, and begin to heat at the forward end. Collect several bottles full of the gas.



31. Arrange a chalk cup on a wire stand about 3 inches high, as in Fig. 8, and set it on a plate containing water. Select a piece of charcoal the size of a small marble and by means of a pair of tongs, or a piece of wire, hold it in a flame until it is ignited, then lay it on the chalk and quickly lower over it a bottle of oxygen. This may be done by covering the mouth of a bottle with a wet glass plate, inverting the bottle, bringing it just above the cup, slipping the plate to one side and lowering the bottle until its mouth dips into water.

When combustion ceases, lift up the bottle and cover its mouth with a glass plate at once, then pour a little lime water into the bottle and shake it.

32. Use the chalk cup, as before, but put a shaving of phosphorus on it (see note 4, page 2). Ignite the phosphorus by touching it with a hot wire and lower a bottle of oxygen over it so that the mouth of the bottle will dip into water. Let the bottle stand until the white fumes disappear, then slip a glass plate under the bottle and quickly turn it mouth upwards without letting the water run out of it. Test this water with blue litmus.

Repeat this experiment, but instead of igniting the phosphorus, let it stand in the oxygen for a day or two.

Is there chemical action?

Repeat the experiment, but use ignited sulphur instead of

phosphorus.

33. Coil about six inches of magnesium ribbon in a spiral about a piece of glass tubing, then slip the ribbon off the tube and attach one end to a piece of wire, ignite the other end and lower it into oxygen. Boil a little of the resulting white powder with water and test the water with red litmus paper.

34. Make some steel wool into a tight roll about the size of a lead pencil and two inches long, ignite the end of it close to the mouth of a bottle of oxygen and quickly

lower it into the bottle.

When there is no further action, examine what is left of the wool. Shake the contents of the bottle with a little water and test with litmus.

This experiment may be varied and made more brilliant by using a piece of steel watch spring or a strand from a small wire rope. In either case the metal should be filed bright for about an inch from one end; then, that end heated, dipped in flowers of sulphur, the latter ignited, and thrust into the oxygen. In this case the litmus test must be omitted, for the vapour of burning sulphur would interfere with the result. There should be a layer of sand or powdered chalk on the bottom of the bottle.

Experiments 31-34 are intended to show the effect that oxygen has upon the rate and intensity of combustion.

How would you express the result of your observations? What leads to these conclusions? Does oxygen form compounds with any of the burning substances? Upon what facts do you base your answer? How was litmus affected by each?

35. A bottle filled with oxygen is closed with a tightly-fitting one-holed rubber stopper that has a glass tube in it, as in Fig. 9. A little strong solution of pyrogallic acid is

poured into the bottle, then half as much solution of sodium hydroxide; the stopper is at once put in and the whole

inverted and allowed to stand so that the end of the tube dips under water in a dish.

Is there any indication that oxygen

Is there any indication that oxygen is soluble in this liquid?

36. Repeat Experiment 21, but attach the nozzle to the tube containing the smaller quantity of gas and direct the gas, as it escapes, against a glowing splinter.

Does the gas itself burn with a flame? 37. Attach a delivery tube, bent as in Fig. 10, to a hydrogen generator, and attach a burner to the outer end of it. Light the escaping gas when it is safe to do so, and invert a dry bottle of oxygen

over the flame.

Fig. 9

What is formed during the combustion? With what is the hydrogen uniting?

38. Put into a eudiometer over water or mercury about 12 cm. of hydrogen and read the volume of the gas. Add about 4 cm. of oxygen and again note the volume. Press the mouth of the eudio-

meter down against a rubber pad under water, or mercury, and pass a spark between the points.

oxygen taken?

What was the reduction in volume? Why was there a change in the volume? What became of the gas that disappeared? How does the reduction of volume compare with the

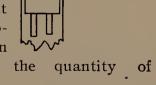


Fig. 10

It is now necessary to find out what gas remains. Pass

oxygen into the eudiometer until the quantity of gas in it is increased nearly, but not quite, one-half. Then try if the mixture will unite, as before, but note the readings at each step.

Of what did the residue of gas from the first explosion consist? How much oxygen ceased to exist as gas, in the first operation; and how much in the second one? What quantity of hydrogen united with the oxygen the first time, and what the second time?

Repeat the experiment, but use nearly equal volumes of the two gases; note the readings on the scale.

How does the reduction in volume compare with the quantity of hydrogen taken?

Pass hydrogen into the eudiometer until the quantity of gas in it is about doubled, then try if combination again occurs, when the spark is passed.

What was the residue in this case? Tabulate the result of the experiment thus:

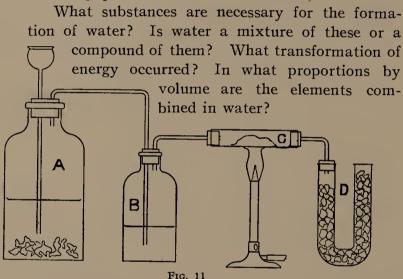
Hydrogen Present.	Oxygen Present.	Volume of Mixed Gases.	Residue after Ignition.	Went into	Oxygen that Went into Combination.		

For every one volume of oxygen that went into combination the first time, how many volumes of hydrogen combined with it? For every two volumes of hydrogen that disappeared as gas in the last trial, how many volumes of oxygen combined with them? In what proportions by volume do

oxygen and hydrogen combine? What hecame of the gases that united?

Note.—The electric spark in this experiment is simply a convenient way of raising the temperature of the gases within the tube to the point of ignition. The heating effect is the important thing, but the fact that the heat was produced by the agency of the electric current is of no significance whatever. A blazing splinter would have served the same purpose.

Sum up the results of the last three experiments as answers to the following questions:



The foregoing determination of the composition of water is known as the volumetric (volume-measure) method; but a gravimetric (weight-measure) method may also be employed.

39. Fit up the apparatus as shown in Fig. 11. A is a hydrogen generator, B is a bottle of strong sulphuric acid through which the hydrogen bubbles slowly in order that it may be freed from moisture, C is a hard glass tube containing copper oxide, D is a U tube filled with lumps of fused calcium chloride, or of caustic soda.

Observe the following precautions:

- (1) Weigh the tube C with the oxide in it.
- (2) Weigh the U tube and its contents.
- (3) After all air has been driven out of the generator, attach the tubes, as shown, and heat the oxide to redness.
- (4) After the black powder has been changed to red let the tube cool, disconnect the apparatus and weigh the same pieces as before.

The object of the U tube is to intercept any water that might be passing out of the apparatus.

How much weight has the tube C lost? How much has D gained? What passed into C? What passed into D?

The object of this experiment is to determine the relative weights of oxygen and hydrogen in water. To what conclusion do your observations lead?

CHAPTER VIII

STUDY OF AIR

40. Repeat Experiment 37, but let the inverted vessel be filled with air instead of oxygen, and let its mouth dip under water. The instant the flame dies out, disconnect the hydrogen tube to prevent that gas from entering the vessel.

Was water formed during the combustion? Does the remaining gas support the combustion of a splinter?

The object of this experiment is to learn something of the composition of air. What have you found out regarding that matter: (1) As to the number of components; (2) as to the characteristics of them?

- 41. Select a long glass tube, closed at one end, such as a eudiometer; measure its length if it is not graduated; wet it inside, then drop into it a freshly cut shaving of phosphorus, which should adhere to the wet glass near the closed end; stand the tube mouth downward in water for a couple of days. Note the temperature of the room, and look up the results of Experiment 32. When the water ceases to rise in the tube, bring the remaining gas to atmospheric pressure by depressing the tube in water until the level of the latter is the same inside and out; make any necessary correction for change of temperature, and calculate the percentage of gas that has disappeared. If this result is less than 20.3, or greater than 21.3, the experiment should be repeated. In any case, it is well to make a number of determinations and take the average of the percentages obtained. This method depends on the fact that phosphorus may combine with the oxygen in the air without ignition, and the compound formed is soluble in water. When phosphorus burns, as in Experiment 32, the enclosed air becomes heated, and as it expands, some of it is likely to escape, so that the measurement is then only approximately correct.
- 42. Pass into a eudiometer over water about 15 cm's of air and half that much hydrogen, read the volumes of air and of the mixed gases, then with proper precautions pass the spark between the points. Read the volume of the residue.

Repeat this experiment several times, tabulating the readings as in the following scheme. Calculate the percentages and average the results. This is an excellent exercise for a class to practice, in groups of three or four members each. All readings should be taken at atmospheric pressure,

Determinations	1	2	3	4	5	6
Volumes of air taken .						
Volumes of mixed gases						
Volumes of residue						
Volumes of gas that disappeared						
. Volumes of oxygen present						
Percentage of oxygen in air				<u>l</u>		

Average percentage of experiments In what proportions by volume do oxygen and hydrogen combine? What part of the decrease in the volume of the mixed gases was due to oxygen going into combination with hydrogen? Why should the volume of hydrogen taken be at least half that of the air in the tube?

CHAPTER IX

CONSERVATION OF MASS

43. Put about 2 grams of lead acetate into a large tt. and boil it with about 30 c.c. of water. Let the liquid stand until it is cool, then pour off 20 c.c. of the clear solution into a clean bottle. Put about a gram of potassium iodide into another bottle with about 20 c.c. of water.

Place both bottles on one scale-pan of a balance and an equal weight on the other one. Then pour the contents of one bottle into the other and put the two bottles on the scale-pan.

Was there any alteration of weight? Was there any new matter formed? Why did the weight not change when yellow solids were produced? Was there a change in the quantity of matter?

44. Fit a large flask with a one-holed stopper through which passes a glass rod long enough to reach nearly to the bottom. Put a little pile of sand or of powdered chalk on the middle of the bottom of the flask inside, and a shaving of phosphorus on the pile. Use vaseline to make the stopper and rod air-tight when they are in the flask, and have the rod just long enough to reach the phosphorus when the stopper is in place. Balance the whole on a scale. Take out the stopper, heat the lower end of the rod, then replace the stopper so that the hot rod will ignite the phosphorus.

What substances were present at first? What are now present? What do the weights indicate regarding the quantity of matter present before the phosphorus burned and after? Turn the flask mouth downward in water and take out the stopper.

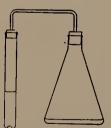


Fig. 12

Has the volume of matter that was in the flask changed? Why?

45. The test tube (Fig. 12) is half-filled with hydrochloric acid, diluted by 20 parts water to 1 of acid. The flask contains a strong solution of caustic soda, to the depth of a quarter of an inch, with some solid pieces of the same substance in it.

The flask and tube are connected by a bent tube that gives communication between the two and by a fine

wire that serves to suspend them to the hook of a balance beam. A piece of calcite or marble weighing about half a gram is laid on one of the stoppers and the whole carefully weighed. Then the stone is dropped into the tt. and the stopper which should be coated with vaseline is quickly replaced.

> How could a change in the quantity of matter be detected? Was there any change in the quantity of matter?

CHAPTER X

OXIDATION AND REDUCTION

46. Powder some red iron rust in a mortar, dry it well and put it in a hard glass tube such as the one used in Experiment 6. Heat it to redness and pass a current of dry hydrogen over it, at the same time hold a cold, dry bottle over the outlet of the tube. After heating for a few minutes let the tube cool and examine its contents. Draw a magnet through the powder.

What passed into the tube with the rust? What passed out? What remained in the tube? What chemical action went on?

Compare Experiments 4, 6, 8 and 22.

CHAPTER XI

SULPHUR

47. Fill a tt. about one-fourth full of yellow flowers of sulphur and heat it. Notice changes in colour and in state. When the sulphur boils allow the vapour to pass into a cold, dry flask.

In what condition is the sulphur when it condenses from the vapour? Will the vapour burn at the mouth of the tube? When sulphur burns, what chemical action goes on?

When the sulphur has boiled for two or three minutes,

pour it into cold water.

What appearance had the vapour that passed from the molten sulphur as it fell through the air?

Compare the substance in the water with the sulphur originally put into the tube in regard to appearance and physical properties. The black, waxy form is *plastic sulphur*.

48. Burn a little yellow sulphur in a chalk cup in air. Notice the colour of the flame; cautiously smell the gas given off; hold a slip of moist blue litmus paper in the gas. Treat some of the black material from the last experiment

in the same way.

Is the black substance sulphur? Why? When sulphur burns in air in a closed vessel there remains a mixture of two gases; what are they?

49. Boil some copper clippings with sulphuric acid and collect the gas that comes off by leading it downward

into a dry vessel.

Is this gas the same as that formed by burning sulphur? Is it soluble? Will it burn? Will it support combustion? Make a mixture of about two volumes of this gas and one of oxygen and try if a blazing splinter will cause them to combine?

SULPHIDE OF HYDROGEN

Note.—These experiments should be made in a room that can be well and quickly ventilated, or in a fume chamber.

50. Drop a piece of iron sulphide, about as big as a grain of wheat, into a little hydrochloric acid in a tt.

Smell *cautiously* the escaping gas. Dip a strip of white paper into lead acetate solution and expose it to the gas. Moisten the surface of a clean silver coin and expose it to the action of the gas. Test the gas with moist litmus paper, both red and blue.

51. Use a hydrogen generator, but put into it some pieces of iron sulphide and either hydrochloric or sulphuric acid diluted one-half with water. Iron sulphide is formed when iron is heated with sulphur out of contact with air; it was produced in Experiment 10, when the mixture was heated in the tube.

Collect a bottleful of the gas over water, smell a handful, hold the mouth of the bottle to a flame, smell the gas cautiously that results from the combustion.

Is there sulphur present? What appearance had the gas when collected in the bottle? Describe the burning of the gas.

When all air has been driven out of the generator, attach a fine burner to the delivery tube and ignite the gas.

Is moisture given off? Hold a cold glass in the flame for two or three seconds. What does this experiment show regarding the composition of hydrogen sulphide?

52. Mix this gas with oxygen in the proportions of two volumes of the gas to three of oxygen and hold the mouth of the vessel to a flame.

Compare the result as to rate of reaction and residue with that obtained in the previous experiment, when the gas in the bottle was burned.

53. Dissolve in separate tubes a little each of copper sulphate, lead acetate, arsenious oxide, tartar emetic, zinc sulphate, iron sulphate and silver nitrate. Add a few drops of hydrochloric acid to all except the last three,

and to them add ammonia. Pass hydrogen sulphide, either as a gas or as a solution of the gas, into each. Note the colour of the precipitates. This treatment with hydrogen sulphide is an important process in chemical analysis.

CHAPTER XII

SODIUM

Refer to Experiments 23-4.

54. Examine a piece of sodium that has a freshly-cut surface, note its appearance, hardness, and whether it is altered by exposure to air or not.

55. Take a piece of sodium of about .1 gram in weight, cut it into six or eight pieces and drop these separately on about 10 c.c.'s of water in an evaporating dish. Rub a drop of the resulting liquid between the finger and thumb.

What does it feel like? Mix a drop of the liquid with four or five drops of water, then taste it. Put a strip of red litmus paper into the liquid. The soapy feeling, the peculiar salty taste, and, especially,

of Alkaline substances.

56. Dilute 10 c.c.'s of hydrochloric acid to 50 c.c.'s, then pour it into a burette and let it run through quickly until the nozzle is full of fluid. Make the solution from the last experiment blue with litmus, then let the acid run into it slowly, and with constant stirring until the blue is *just changing to red*.

the turning of red litmus paper blue, are characteristics

What taste has the liquid now? Has it the same soapy feeling as before?

Let another drop of the acid solution fall into the dish.

What is the result?

Repeat the experiment, using other acid solutions separately.

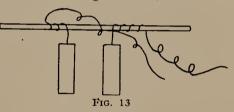
In what respect do the results agree? In what respect do they differ?

CHAPTER XIX

IONISATION

57. Electrolysis.—Use a battery of at least six dry cells, or one of equivalent strength. A pair of platinum plates, such as are used in electrolytic apparatus, with wires attached, should be fastened to a glass rod, as in the

diagram; the faces of the plates should be parallel and held an inch apart; a galvanoscope should also be connected in circuit to show whether

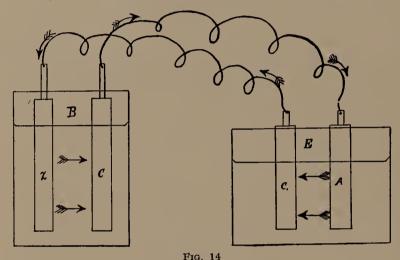


or not a current is passing. Introduce a lump of dry copper sulphate into the circuit. This may be done by cutting a wire and pressing the cut ends against the lump. Substitute for copper sulphate, potassium nitrate (saltpetre), iron sulphate (copperas), calcium chloride, sodium hydroxide and sodium carbonate (washing soda).

Dissolve the solids just used in separate beakers of water, then attach the battery wires to the plates on the glass rod, dip the plates first into pure water, then, successively, into the solutions.

Are the salts, when in the solid form, electrical conductors? Are their solutions conductors? Is water a conductor? Salts, when in solution, have what properties that they do not possess in the solid state?

Make at least three solutions of different strengths of any one of the above salts, and determine whether or not the resistance to the current varies in solutions of different strength; and, if so, whether it varies directly or inversely with the strengths of the solution. Try if glycerine, concentrated sulphuric acid, solution of sugar, gelatine boiled in water, or coal oil, will conduct an electric current.



58. Make a solution of copper sulphate, put it into a beaker and dilute it until it is light blue in colour; slip the platinum plates an inch apart on the rod and dip them into this solution, then connect the battery. Watch the plates. Remembering that a current leaves a battery by the carbon pole and returns to the zinc pole, determine whether the copper is deposited on the plate (electrode) by which the current enters the electrolyte (anode), or the one by which it passes out (cathode). When the solution becomes decolourised, evaporate a few drops of it to dryness. Reverse the current in the same solution and notice the plates. Use the clean plate for a cathode and

a piece of copper wire for the anode, with some of the original blue solution for the electrolyte.

Does the decolourised solution still contain copper sulphate? Where is the copper? Does the copper wire change when used as an anode? Does copper move through the liquid? When a copper anode is used, is the liquid decolourised? What is the source of the copper deposited on the cathode: (1) When platinum plates were used; (2) When a copper anode was employed.

59. Make a solution of potassium nitrate, fasten strips of litmus paper, both blue and red, to the platinum plates and place them in the liquid.

What does the litmus show? Why does potassium not gather on one plate as the copper did? (Refer to Manual, Experiment 23.) Is the fluid about the two plates the same? Which plate shows concentration of alkali? Has there been any movement of matter in the solution?

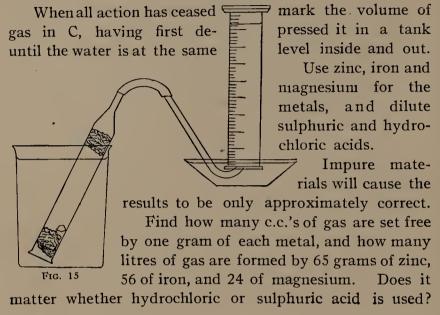
CHAPTER XX

EQUIVALENCE

60. Compare the quantity of hydrogen set free by definite weights of various metals acting on dilute acids, as follows: A is a beaker containing the acid. B is a test tube drawn out at the bottom to a quarter-inchitube, and having in it two loose plugs of asbestos fibre. C is a gas cylinder, or more conveniently, a graduate marked in c.c.'s.

Weigh accurately a piece of zinc of about .6 or .7 of a gram in weight, a piece of iron wire of about the same weight, and a piece of magnesium of about .3 of a gram. Lift the tube B out of the acid, let it drain, remove the

mouth plug, drop in the metal, replace the plug in the tube and the tube in the acid.



CHAPTER XXIII

NITROGEN

61. Attach a piece of lead to the underside of a large cork to steady it when floating, place on the cork a small plaster of paris or chalk cup containing phosphorus, and float the whole on water. Ignite the phosphorus and cover it with a bell jar or inverted bottle whose mouth dips under water. Let the whole stand until the white fumes entirely disappear. Slip a glass plate under the vessel and invert it without letting any water escape from it. Test the gas that remains in the vessel with moist litmus paper, with a burning splinter, and with a jet of burning hydrogen.

What compound did the phosphorus form when it burned? What purpose did the phosphorus serve in this experiment? What advantage comes from the solubility of the white fumes? Compare Experiments 35, 41, 42.

CHAPTER XXIV

NITRIC ACID

(PRECAUTION.—Do not allow nitric acid or oxides of nitrogen to touch the skin or clothing.)

62. Put into a retort some nitrate of either potassium or sodium, add sulphuric acid, pass the neck of the retort into a flask that rests in water and heat the mixture in the retort just sufficiently to keep bubbles of gas escaping freely into the flask.

Describe what happens.

Definition.—The process here employed for separating and collecting the acid is called Distillation. The yellow liquid—distillate—that collects in the receiver is nitric acid. When sufficient acid has been collected, heat the liquid in the retort more strongly. The dense brown fumes that appear are not vapours of nitric acid, but an oxide of nitrogen, due to the decomposition of the acid by excessive heat, as will presently be explained.

63. Put a few drops of the acid into an evaporating dish and warm it, then hold a piece of ignited charcoal, in a pair of tongs, *close to the surface* of the liquid, but not touching it.

Is there any appreciable change in the rate of burning of the charcoal when first brought near the acid? Is there any change afterward? Given that combustion of charcoal is chemically a case of oxidation, how is the more vigorous burning accounted for?

64. Again put a few drops of acid in the dish and place a shaving of phosphorus in it. Cover quickly with a glass plate and watch what happens.

What was the first evidence of chemical action? Did any brown fumes appear in this case? Compare the rate of burning of phosphorus in air and in contact with nitric acid.

- 65. Put a little of the acid in a tt. and boil it. The brown fumes that are formed consist of oxides of nitrogen mixed with a little steam. The colour is due mainly to nitrogen peroxide, NO_2 . Nitric acid, when heated to about 85° C., begins to undergo decomposition, thus, $2HNO_3\rightarrow H_2O+2NO_2+O$. The NO_2 is itself a very unstable compound, readily becoming reduced to NO. It follows that nitric acid acts as a powerful oxidising agent. An equation to indicate the quantity of oxygen set free is $4HNO_3=2H_2O+4NO+3O_2$.
- 66. Pour a few drops of the prepared acid into a tt. and insert loosely in the mouth of the tube some thin pine shavings; boil the acid and watch what happens when the brown gas rises to the shavings.
- 67. Put a little ordinary (commercial) nitric acid into a tt., dilute it one-half with water and drop in a piece of zinc. If gas does not come off warm the liquid.

What appearance has the gas? Determine if it is hydrogen.

Repeat the experiment, using copper instead of zinc.

What evidence is there that chemical action goes on? What causes the liquid to turn blue?

68. Burn a little sugar on a piece of mica. Notice the rate of the combustion. Make a mixture of equal parts

of powdered sugar and potassium nitrate (saltpetre) and burn it, as before, but hold the mica in a pair of tongs. Take as much gunpowder as will lie on a five cent piece and ignite it with a match held in a pair of tongs. Gunpowder is a mixture of sulphur, charcoal and potassium nitrate ground together.

How do the rates of burning compare? To what is the difference due? Could the gunpowder possibly burn at the rate it does if the oxygen came from the air? Where does the oxygen come from that enters into the combustion of powder enclosed in a gunbarrel or in a hole in a rock?

CHAPTER XXV

OXIDES OF NITROGEN

69. Drop some copper into strong nitric acid and collect the escaping gas over water by displacement. When the generating tube is filled with the dark brown fumes, take out the stopper and quickly turn the tube mouth downward in water. After the water ceases to rise, lift the tube until a bubble of air enters and then replace its mouth under water. Repeat this. Fill a dry tt. with the brown gas and place it mouth downward in water.

Was there one gas or two that came off from the generating tube? What is the reason for your answer? Was the colourless gas that was collected over water, air, or hydrogen, or oxygen? Why? When air was admitted to the colourless gas, what took place? Was there a chemical change when air was admitted? Why do you think so?

70. Heat some ammonium nitrate in a test tube fitted with a delivery tube so that the gas given off may be col-

lected over water. When the salt melts, graduate the heat so as to keep a gentle stream of gas coming off. Test the gas (1) with a glowing splinter; (2) a blazing splinter; (3) burning phosphorus.

Will the gas burn? Will it support combustion? How does it affect litmus? Is it soluble?

The gas produced in this case is nitrous oxide, according to the equation $NH_4NO_3 \rightarrow N_2O + 2H_2O$.

CHAPTER XXVI

AMMONIA

71. Boil a strong solution of ammonium chloride with lime and allow the escaping gas to pass into a dry inverted vessel that has a strip of moist red litmus paper across its mouth. Test the gas for solubility, combustibility, power of supporting combustion, and weight as compared with air. Before determining the weight, the gas should be dried by passing it among lumps of fused calcium chloride, not through sulphuric acid. Test the dry gas with dried red litmus. Compare with the result when moist litmus is used

Hold a delivery tube, from which ammonia is escaping freely, about two or three inches above the top of a gas or lamp flame.

Will ammonia burn? Will it continue to burn if removed from above the flame?

72. Pass ammonia and oxygen in about equal proportions into a bottle held mouth downward; when the bottle is full of the mixture try to burn the gas.

What is the result?

73. Boil about ten c.c.'s of strong ammonia solution in a thick walled flask, and after the vessel is completely

filled with ammonia gas, invert it, placing its mouth under water. If the water does not almost fill the flask the experiment should be repeated.

Nessler's test for ammonia is prepared as follows: Dissolve 3 grams of potassium iodide in 25 c.c. of water; add to this gradually, with stirring, a cold saturated solution of mercuric chloride (corrosive sublimate) until the precipitate entirely disappears, then add a solution of 15 grams of potassium hydroxide in 15 c.c. of water. Dilute the whole to 100 c.c. with cold water, allow the turbidity to settle, then decant the clear liquid. A little of this when added to a liquid should show yellow or red discolouration if ammonia is present.

CHAPTER XXVII

RATE OF CHEMICAL REACTIONS

74.(a) Dilute a little nitric acid with ten times its own volume of water, make a second portion one-half water and use a third portion full strength. Drop a small piece of marble into each.

Does chemical action go on in all three liquids? In what one is the action most energetic, as shown by the disappearance of the marble?

(b) Dilute some nitric acid one-half with water and divide it into two parts; heat one part to boiling, then drop a piece of marble into each.

In which fluid does the more vigorous action go on?

(c) Weigh out two equal quantities of marble, one as a lump, the other powdered. Put these separately into two similar portions of dilute acid.

Which disappears more quickly, the lump or the powder? How is the surface area of the marble changed by powdering it? Why should there be a difference in the rate of chemical action?

75. Dilute some hydrochloric acid with ten times its volume of water and pour equal quantities into each of two test tubes. Select two similar crystals of sodium carbonate; dissolve one in as little water as possible, then put the dry crystal into one of the tubes containing acid and pour the solution into the other.

What is noticeable about the rates of chemical action in this case? What effect has dissolving a mass on its state of division? What comparison is there between the quantities of gas set free in the two cases? What comparison of lengths of time during which the gas escapes?

76. Bring into contact with each other a dried crystal of potassium iodide and one of bichloride of mercury (corrosive sublimate), notice if there is any discolouration. Rub them to powder in a very dry mortar and examine the mixture, still watching for change of colour. Let a drop of water fall on the powder. Make a little solution of each substance separately, and pour the solution of the potassium iodide into that of the mercuric chloride.

What effect has powdering and mixing two substances, on the number of points of contact between them, as compared with the number of such points before powdering? Provided chemical action occurs between the substances in the solid state, how would it be affected by increasing the number of surface contacts? What would be the effect on the rate of chemical action, of powdering and mixing the masses? What relation does dissolving bear to grinding to powder in respect to

separation of parts of a mass? What relation is there in respect to points of contact when two solutions are mixed, as compared with powdered solids?

77. Make a solution of about half a gram of iodide of potassium in 20 c.c. of water. Make starch paste by pouring about 100 c.c. of boiling water on about one gram of powdered laundry starch and stirring the mixture thoroughly. A drop of the iodide solution should produce no discolouration when added to a little of the starch paste. Divide the iodide solution into two parts in separate bottles, wrap one in dark paper and put it away in a drawer, in the dark; set the bottle containing the other part of the solution where it will be exposed to bright sunlight. Test the contents of both bottles from time to time by letting a drop of the fluid from each fall into a little of the starch. A blue colour produced in the latter indicates free iodine.

The point to be determined is whether or not iodine is set free; and, if so, in which case the reaction first takes place.

What conditions probably produced the result observed?

CHAPTER XXVIII

HYDROGEN CHLORIDE

78. Mix a large spoonful of common salt (sedium chloride) in a flask with sulphuric acid, heat the flask and allow the gas to escape through a delivery tube. Cautiously smell a handful of the gas. Collect the escaping gas by leading it downward into a dry vessel. Test it for solubility and combustion. Find if it is soluble in

coal oil, benzine or gasoline. Fill a dry bottle with ammonia gas, and place the mouth of the delivery tube in it. When the bottle is full of white fumes cork it and let it stand.

What appearance has this gas when it escapes into the air from the delivery tube? Blow the moist breath across the jet of escaping gas. What appearance has it when collected in a vessel? What became of the white fumes in the ammonia bottle? Was the white cloud composed of gaseous, of liquid, or of solid particles?

79. Make solutions of the gas in coal oil and in water. Drop some powdered marble into each.

Is there a reaction in either case?

80. Make a solution of the gas in water and add a drop of silver nitrate solution; then add ammonia until the liquid is alkaline. Repeat the experiment, using instead of hydrogen chloride, sodium chloride (common salt), ammonium chloride and iron chloride separately to form the solutions.

Expose the white precipitate in each case to the action of bright light in a window for a few minutes.

This experiment shows how the presence of any soluble chloride may be determined.

State clearly the effect produced—(1) by the silver nitrate; (2) by the ammonia; (3) by the light. How may a solution of hydrogen chloride be distinguished from that of any of the other chlorides mentioned?

81. Make a solution of hydrogen chloride in water, and another one in coal oil. Use carbon pencils as electrodes and determine, 1st, if the oil solution is an electrolyte, then if the water solution is one. Make sure that the electrodes are dry when immersed in the oil solution.

Is water alone an electrolyte? Is there any evidence that either solution has free ions in it?

Shake some of the oil solution with water, let the water settle to the bottom, then place the dried electrodes in the oil and gradually lower them into the water.

What conclusion does the observation warrant?
Put some powdered marble into the solutions of hydrogen chloride in water and in oil.

What difference of result is observed?

CHAPTER XXIX

CHLORINE

Note.—Make all experiments with chlorine in a ventilated cupboard or fume chamber.

82. Heat together some manganese dioxide and hydrochloric acid in a flask, collect the gas that comes off by leading it downward into a vessel whose mouth is covered with a glass plate or a piece of wet card. The greenish yellow gas is Chlorine.

Is chlorine soluble? Does it affect litmus? Does it burn? Will it support the combustion of charcoal? Place a piece of phosphorus on a chalk cup in a jar of the gas. What occurs?

Let the gas bubble through water in a bottle, and use the solution, when possible, in determining the properties of chlorine. The gas may be collected over strong brine.

83. Half fill a test tube with hydrogen, pass in an equal volume of chlorine, test the mixture with litmus paper, then hold the mouth of the tube to a flame, test again with litmus. Pass a jet of burning hydrogen into a jar of chlorine. Test with litmus the product of the combus-

tion after the chlorine disappears. Fill two stoppered bottles with a mixture half chlorine and half hydrogen; expose one to the light of a window, and keep the other away from bright light. Test the contents from day to day.

84. Pour some saturated solution of chlorine into each of two stoppered bottles, put one where it will be exposed to bright light and the other in a dark place; test these with litmus paper once a week to find if either undergoes any chemical change. Note what that change is, which shows it first, and the length of time before it can be detected in the other.

What condition is different in the two cases? How is the difference in rate of reaction accounted for?

- 85. Compare the appearance of red lead with that of lead peroxide. Shake up with water in a tt. as much of the former as will lie on a five cent piece; let chlorine bubble through the water until a strong solution of it is obtained, then let the whole stand for half a day. Test the liquid for acidity.
- 86. Use two stoppered bottles; put into one some pieces of coloured cotton or linen that have been well dried, and into the other some similar pieces that have been dipped in water; then fill each bottle with chlorine that has passed slowly through strong sulphuric acid to remove all moisture from the gas. Close the bottles and compare the results.

What condition varied in the two bottles? To what is the difference in effect due?

87. Mix some "Chloride of Lime" (bleaching powder) with water in a tt., add a little dilute sulphuric acid, and determine if chlorine is given off. Dip some pieces of inkstained paper and of coloured cotton in some water

that has enough bleaching powder in it to coat the fabric thickly, then remove the pieces to a vessel containing very dilute acid. Try whether or not a similar result would be obtained by adding acid to the powder and water while the dyed material is in it.

88. Determine whether a solution of chlorine will yield results similar to those obtained in Experiment 80.

If so, how may chlorine solution be distinguished from solutions of hydrogen chloride and of ammonium chloride?

89. Heat a mixture of nitric and hydrochloric acids in a tt. that has a strip of litmus paper across its mouth.

Is chlorine given off?

Float a small piece of gold leaf on a few drops of nitric acid in an evaporating dish and let it stand for a couple of hours. Do the same with hydrochloric acid. Afterwards pour the two together.

Does either acid alone affect the gold? Does the mixture? How may this result be explained?

BROMINE AND IODINE

Note.—These experiments should be made in a fume chamber.

90. Mix some potassium bromide with manganese dioxide and pour the mixture into a test tube containing sulphuric acid. By means of a delivery tube, lead the generated gas downward into bottles. When two or three have been filled, cork them and pass the rest of the gas into water in a bottle. Carefully avoid inhaling this gas.

What appearance has the gas? Determine whether it is soluble or combustible? What effect has it on litmus solution? What effect has it on pieces of coloured cotton? Add some solution of

the gas to silver nitrate solution, then make the whole alkaline with ammonia. Lower a piece of phosphorus in a chalk cup into a bottle of the gas. Pass some of the gas into a tt. surrounded with ice. This reddish brown gas or liquid is bromine.

91. Make a solution of potassium bromide, add some chlorine solution to it. Heat the liquid.

What gas passes off? Pass the gas into starch made by stirring some laundry starch with boiling water to a thin paste. Bromine, when present in quantity, stains starch yellow.

- 92. Put about 5 c.c. bromine solution into a bottle and add a thin shaving of phosphorus. After the liquid loses its colour pour it into a tt. fitted with a delivery tube. Test the liquid for acid properties, then heat it and pass the escaping gas into a little cold water. Test this liquid for acidity, then drop in a little chlorine solution. The acid liquid is a solution of hydrobromic acid.
- 93. Heat a little potassium iodide with manganese dioxide and sulphuric acid in a tt. fitted with a cork and a straight delivery tube all held vertically. Drive the black deposit into the delivery tube by heating from below.

What colour is the vapour? What appearance has the condensed solid? This solid is *iodine*. Scrape off a little of the iodine and shake it with a few drops of water. Is it soluble? After standing for a day add a little alcohol to part of the solution that contains undissolved iodine, and a little solution of iodide of potassium to another part. What is the conclusion regarding the solubility of iodine in water, in alcohol, and in potassium iodide solution?

94. Drop a very small piece of solid iodine into some boiled starch. Will iodine solution act similarly? The

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discolouration of the starch is a test for the presence of free iodine. Drop a crystal of iodide of potassium into some starch. After it has dissolved, add a few drops of chlorine solution to some of the starch, and to another portion of it add a little bromine solution.

What does this indicate about the relative combining powers of chlorine and iodine for potassium? 95. Put about 5 c.c. of iodine solution, made with alcohol, into a tt., and add a thin shaving of freshly-cut phosphorus. Let the whole stand until it is colourless, then pour off the liquid and test it for acid properties. Heat this liquid in a tt. fitted with a delivery tube and pass the gas that comes off into about 5 c.c. of cold water. Test this water with blue litmus paper; afterwards shake it with a little starch and add a drop of chlorine solution. The gas that passed over was hydriodic acid.

Compare the three elements, chlorine, bromine and iodine, in regard to (1) their preparation; (2) their conditions at ordinary temperature; (3) their colours; (4) their chemical activity as measured by their action on litmus and by their power of displacing one another; (5) the action of phosphorus upon their solutions; (6) their solubility.

CHAPTER XXX

CARBON

96. Put some pine shavings in a hard glass tt., insert a perforated cork in the mouth of the tube, heat the tube, at first slowly, then to redness.

What change do the shavings undergo? What passes out of the tube? Will the gaseous substances burn?

After the shavings have become charred to a black mass, let the tube cool and turn the contents out on a piece of paper.

Will the black residue burn? Why did the shavings not burn in the tube?

Heat as much white sugar as can be heaped on a ten cent piece, in a flame on a thin sheet of mica or of iron. When the mass turns quite black and vapour ceases to come off, let the residue cool.

Will the black substance burn?

That which remains from the treatment of both shavings and sugar is **Carbon**, mixed with a little mineral impurity which remains as *ash* when the carbon is burned. The residue from the wood is known as *charcoal*, that from the sugar is commonly called *coke*.

97. Close the holes at the base of a Bunsen burner to produce a luminous flame, and hold a tt. filled with cold water in that flame near the top. If gas is not available, an alcohol lamp will serve the purpose, provided a little spirits of turpentine, or dissolved camphor, be poured into the alcohol.

What is deposited on the tube? Scrape a little of this material off on paper and examine it. What was the source of the black substance?

Tear a piece of soft paper in two, saturate one part with spirits of turpentine, then burn them separately, the dry one first.

What substance is formed in one case, but not in the other?

The black deposit on the tube and the black powder (smoke) that passed off from the saturated paper are both *lampblack*, a form of carbon commonly called *soot*.

98. Hold a small piece of graphite in a flame for several minutes. If the naturally occurring mineral is not

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available, a piece of the "lead" out of a soft black lead pencil may be used.

99. Put the end of a splinter of wood in strong sulphuric acid and let it stay there for a day; then wash it and compare it with a match that has been half burned.

What effect has the acid on wood?

Put a spoonful of white sugar in strong sulphuric acid in an evaporating dish and let it stand for twenty-four hours; then wash it by filling the dish with water and, after the solid matter settles, pouring off the liquid. Repeat the washing a couple of times.

Examine the black residue in regard to taste, solubility and combustibility. Compare the results with those obtained in Experiment 96.

100. Select a four or five-inch tt. about half an inch in diameter, fill it with ammonia gas and hold it mouth downward in mercury in a dish. There should be more than enough mercury to fill the tube. Prepare a piece of soft, well-burned charcoal by paring it so that it will easily pass into the tube, then holding it in the flame until it is glowing on all sides. Notice if the mercury has risen in the tube. Put the charcoal into the mouth of the tube while the latter is still inverted in the mercury.

How does the volume of gas in the tube change? Did the ammonia escape? Is there any indication that it either dissolved in the mercury or combined with it?

When the mercury ceases to rise, lift the tube very gently out of the dish so as not to spill the mercury, turn the charcoal out and smell it cautiously. Heat the charcoal gently in a dry tube to determine if it gives off ammonia.

What became of the ammonia in the tube over the mercury?

CHAPTER XXXI

OXIDES OF CARBON

101. Put about 2 grams of crystals of oxalic acid into a tt. and pour in sulphuric acid enough to cover it; heat the mixture and, by means of a delivery tube, pass the gas that comes off into two inverted bottles filled with a weak solution of caustic soda, and standing in the same liquid. When the bottles are full, let them stand over the solution until no more gas is soluble. Meantime, try to burn the gas passing out of the delivery tube. Lift the two bottles out of the soda solution, cover one with a glass plate, hold the other one to a flame, then test the contents of each with lime water.

Lime water is prepared by stirring lime with water and letting the whole stand until clear. It is a solution of calcium hydroxide, Ca(HO)₂.

Carbon dioxide causes lime water to turn milky (white) in colour.

The gas that burned was carbon monoxide, CO. The gas that turned the lime water milky was carbon dioxide, CO₂. Test the residue from the burning gas with limewater.

What colour was the flame?

- 102. Twist a piece of wire around a small lump of charcoal, and, using the wire as a handle, ignite the charcoal and put it in a bottle of oxygen; cover the mouth of the bottle with a wet card or a glass plate. After a minute or two withdraw the charcoal and shake the contents of the bottle with lime water.
- 103. Put into a hydrogen generating bottle a lump of sodium carbonate (washing soda), and pour on it some hydrochloric acid, diluted with ten times its own volume

of water. Collect the gas that comes off by displacing water from inverted bottles.

Test the gas with lime water, also determine if it is soluble and how it acts when a blazing splinter is thrust into it? Set one of the bottles filled with the gas mouth downward in sodium hydroxide solution. Find whether or not a piece of limestone, calcium carbonate, CaCO₃, may be used instead of the soda?

104. Balance a dry flask and stopper on a scale, then fill the flask with carbon dioxide that has been dried by passing through strong sulphuric acid, or calcium chloride lumps. When the flask is full insert the stopper and put it back on the scale-pan.

105. Set a piece of burning candle in the bottom of a beaker; then try if carbon dioxide will pour downward, as water would, into that vessel.

What conclusions may be drawn from this experiment?

106. Pass a stream of carbon dioxide through some lime water contained in a tube. When the liquid becomes white, divide it into two parts; add a drop of hydrochloric acid to one part, but continue to pass the gas through the other part for ten minutes. After the solution becomes clear, divide it into two parts and pour each into a clean tube. Boil one, add a few drops of ammonia to the other.

What changes were noticeable in the solution while the gas was passing through it? What changes occurred afterwards? Account for these.

107. Fasten a piece of magnesium ribbon, about three inches long, in a slit in the end of a stick. Ignite the magnesium and quickly lower it into a bottle of carbon dioxide. Compare the result with that obtained when a

blazing splinter was used, Experiment 103. After the magnesium ceases burning shake the contents of the bottle with a little water.

How are the black particles accounted for? If combustion takes place only in free oxygen, how may the following facts be explained: (1) Carbon dioxide is 73% oxygen by weight, air is 23%; a splinter will burn in air, but will not burn in carbon dioxide? (2) Magnesium will burn in carbon dioxide, a splinter will not, but both will burn in air?

108. Fill a bottle with carbon dioxide and shake the gas well with a little water, then test the water for acidity. The litmus may have to stand in the solution for some time before it shows a change.

109. Fill a large bottle of at least a quart capacity with exhaled air by breathing into it two or three times through a tube passed to the bottom of the bottle, then shake the contents with about a spoonful of lime water.

Fill the same bottle with water and empty it in the open air, then test the contents again with lime water.

What conclusion may be drawn about the composition of exhaled air?

CHAPTER XXXIII

ACETYLENE

110. Fill a small, wide-mouthed bottle with water and turn it mouth downward in a plate of water, wrap a small piece of calcium carbide in paper and slip it under the mouth of the bottle. When the bottle is about one-tenth filled with the gas that rises from the carbide, lift it out of the water so that air will mix with the gas. After standing for a minute ignite the mixture. Make this experi-

ment three times, but the second time half fill the bottle with the gas; the third time fill the bottle completely.

What differences are there in the burning of the three bottles of gas? In which case was most carbon set free? Why was more carbon set free one time than another? In which case was the combustion most rapid? Why was there a variation in the rate of the reaction? What was the source of the carbon that was given off? How can a colourless gas set solid carbon free from it?

111. Use a stoppered bell jar or a bottomless bottle, fitted with a delivery tube drawn out fine (the nozzle of a blow-pipe is very suitable). Open the clip, press the bottle down in a tank of water until it is full, close the clip and raise the bottle until its mouth is only a little way below the surface of the water. With a pair of tongs or a piece of wire hold some carbide wrapped in muslin under the mouth of the bottle until it is full of gas. Press the bottle down so as to keep the level of the water outside the bottle about two inches higher than that inside, then open the clip and ignite the gas at the jet. When the gas is all consumed,



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refill the bottle with coal gas and burn it in the same way.

Compare the brightness of the two flames. Repeat the experiment with acetylene, but use for a burner a tube of one-eighth of an inch bore. What difference between the flame now and what it was in the former case in regard to: (1) brightness; (2) carbon given off?

112. Fill the bottle used in the last experiment with acetylene. Light the escaping gas at the jet, as before; then slowly lift the bottle out of the water while the gas

is still burning. Hold the bottle so that if a flame flashes out of the lower end it will do no harm.

How is the action that occurs accounted for? Mention three notable points connected with the burning of acetylene.

FLAME

113. Examine a candle flame that is burning steadily and free from the influence of wind currents.



Fig. 17

What colour is the lower part at 1 where it spreads out? What is observed at the parts numbered 2 and 5?

Cut a shield the shape of the flame in the figure, close one eye and hold the shield at such a distance from the other one as to cover the bright part of the flame, but not the margin.

What appearance has the outer fringe of the flame?

Cut several strips of stiff white paper, each about two inches wide; these are to be held horizontally in the flame just long enough to show

charring, but not to become ignited. Hold one in each of the positions marked, 2, 3, 4, 5; number them as they are taken out and lay them side by side in order.

How do you interpret the ring markings? What meaning attaches to ring markings on some papers and not on others? At what parts of the flame were the rings most noticeable? From what part of the flame did the black deposit come? Does a

FLAME 57

candle flame burn uniformly throughout its mass? What message does the paper bear concerning that part of the flame at 2, just above the wick? What relation is there between the rings and the grey portion of the flame?

Close the holes at the base of a Bunsen burner, light the gas and turn the flame down until it burns quite steadily; a height of about two inches is suitable. Explore that flame with slips of paper and find if it corresponds in structure with the candle flame.

114. Fill a tt. with cold water and hold it in the non-luminous (blue) flame of a Bunsen burner or of an alcohol lamp. Repeat the experiment, but use a luminous flame.

What is the deposit on the tube? Hold a cold, dry bottle inverted over a flame for a minute or so. What conclusions do these observations warrant regarding the composition of the burning matter?

115. Project into a non-luminous flame successively, a little finely ground charcoal, lampblack, powdered chalk from an eraser, and very fine iron filings. Also hold in the flame a piece of fine wire.

How does the presence of the solid affect the quantity of light emitted? What is the source of the luminosity of the flame when the solid is in it? A glowing solid is said to be *incandescent*.

116. Make some touch paper by soaking some strips of soft paper in a concentrated solution of potassium nitrate and drying them. Ignite one of these strips and hold it close to the holes in a Bunsen burner in which gas is burning with a non-luminous flame. Burn another strip inside of a clean, dry bottle, held mouth downward.

Is the smoke made up of material that is not gaseous? What is the evidence of this?

117. Hold a piece of fine wire gauze, preferably copper or brass, about two inches above the top of a gas burner, turn on the gas and light it above the gauze. Let the gauze cool, then bring it gradually down on top of a flame. Bring a tt., filled with cold water, slowly up against the side of a flame.

Does the flame touch the cold glass? 118. Put some broken soft coal in the tt. of the apparatus figured and heat it, leaving all tube connections open. Notice the appearance of the vapour that passes out. Try to ignite it. After burning a short time, close the clip so as to drive the escaping gases through the water in the tube. Ignite the escaping. gases at the Fig. 18 other vent.

Examine the brown substance left in the water. What does it appear to be? Pour off a little of the water into a test tube and add some of Nessler's reagent for ammonia. Test the water with litmus.

CHAPTER XL

WATER OF CRYSTALLISATION

119. Put a crystal of copper sulphate in a test tube. Hold the tube horizontally and heat it gradually until it becomes red-hot.

What change occurs in the composition of the crystal? What change is there in its appearance? Add a drop of water to the powder after the tube has cooled.

Repeat the experiment using (1) a piece of crystalline (ice like) carbonate of soda; (2) some of the white powder of carbonate of soda.

Select a piece of bright green ferrous sulphate (copperas or green vitriol) and heat it in the same way.

What conclusion in each case?

If there is opportunity, crystals of these substances may be prepared for the experiment by making, separately, saturated solutions of them, and allowing these to evaporate slowly.

CHAPTER XLIII

SODIUM

120. Cut a piece of sodium weighing .1 gram (about as large as a grain of wheat) into five or six pieces, drop these separately on about 5 c.c. of water in an evaporating dish, covering the dish each time with a glass plate. Test this liquid with litmus. Mix a drop of this solution with five drops of water and taste the mixture. Using a burette, neutralise the water in the dish with dilute hydrochloric acid, 1 to 5. Taste the liquid after neutralisation. Evap-

orate the liquid to dryness, preferably over a waterbath. Taste the solid.

What is left?

Write equations for these reactions. Refer to Chap. XII.

CHAPTER XLIV

CALCIUM

121. Treat a little common, non-crystalline limestone with dilute hydrochloric acid. Repeat the experiment, using crystalline limestone, either marble or calcite.

Is there any difference in the liquids left in the tubes when all gas has passed off? What conclusion about the composition of the two kinds of rock?

122. Lay a small piece of limestone on moist litmus paper, then heat a splinter of that limestone until it glows for two or three minutes.

Has the heated part changed in appearance? Will it affect moist litmus?

123. Treat a little freshly-burned lime with dilute acid, then some lime that has been exposed to the air for several months, and some mortar that has been on a wall for a long time.

What difference in the results observed? Test any gas that comes off to see if it is CO₂.

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